	Experiment title: An X-ray study strain and tilt in In_2O_3 epilayers grown on cubic zirconia (111) and (110) by oxygen plasma MBE	**Experiment number:** **28-01-924**
Beamline:	**Date of experiment:** from: 20th April 2011 to: 26th April 2011	**Date of report:** *Received at ESRF:*
Shifts: 18	**Local contact(s):** Didier Wermeille	

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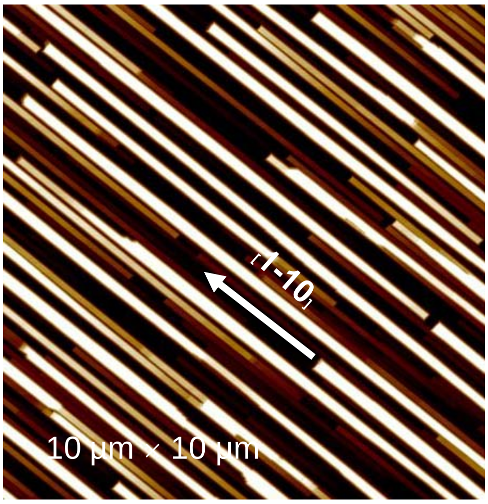
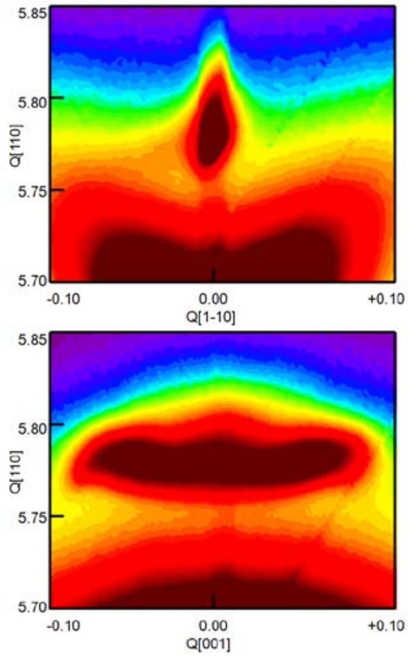
Introduction.

Indium oxide is an important transparent conducting oxide that adopts the body centred cubic bixbyite structure. There has been a recent upsurge of interest in the growth of single crystal thin films of In_2O_3 on face centred cubic Y-ZrO_2 substrates[1-2] owing to the small mismatch of only 1.7% between $2a_s$ for the substrate and a_e for indium oxide, where the a are lattice parameters: the epilayer is under tensile stress. In previous work we have established that indium oxide grows on Y-stabilised ZrO_2(001) surfaces in the form of square micron or nanosized islands[3], whereas on (110) surfaces extended nanorods are obtained. Continuous thin films are obtained on (111) oriented substrates. These differences arise from a sequence of surface energies $\gamma(001) >> \gamma(110) > \gamma(111)$ and the adoption of growth morphologies which promote development of low energy {111} facets. For (001) oriented islands mismatch between the substrate and the epilayer is acommodated in part by a characteristic tilting of the islands[4], as revealed by reciprocal space mapping experiments conducted in-house. In the current contribution we explore the occurrence of strain and tilt in In_2O_3 thin films and nanorods grown respectively on (111) and (110) oriented substrates using reciprocal mapping conducted on the XMaS beamline.

Results and discussion.

Reciprocal space maps for both specular and non-specular Bragg peaks were measured for six samples of thin film In_2O_3: three samples grown on Y-ZrO_2(110) at a series of three substrate temperatures up to 1000 °C; and a series of three samples of different thicknesses grown on Y-ZrO_2(111) at 700 °C.





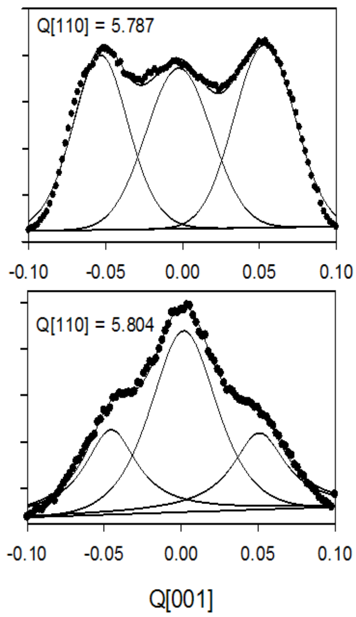


Figure 1 (left hand panel). AFM image of In_2O_3 grown on Y-ZrO_2(110) at 1000 °C.

Figure 2 (central panel). Reciprocal space map for nanorod sample depicted in figure 1 taken around the epilayer (880) reflection. The transverse wavevector transfer is along the $[1\bar{1}0]$ direction in the upper panel (along the rods) and in the [001] direction (across the rods) in the lower panel. Striking anisotropy is apparent. Units are reciprocal lattice vectors of the substrate.

Figure 3 (right hand panel). Transverse slices through the map shown in the bottom panel of figure 5 (i.e. along [001] direction) for two different values of Q[110]. The 3-peak structure is interpreted in terms of a small tilt or rotation of some rods around the $[1\bar{1}0]$ direction along [001] and $[00\bar{1}]$ directions, with untilted rods giving the central peak. More highly strained rods with larger Q[111] show a lower propensity to tilt. By reference to the AFM images we assume it is the thinner and narrower rods that are untilted.

The most interesting results for the (110) oriented samples were obtained for the sample grown at 1000 °C. Atomic force microscopy (AFM) images revealed that this sample had a highly anisotropic morphology with rods of dimensions of up to about 10 μm along the $[1\bar{1}0]$ direction and widths of the order of 20 nm- 200 nm along the [001] direction (figure 1). Quite narrow rods were found to coexist with relatively broader rods. As shown in figure 2 the reciprocal space maps for this sample were also strongly anisotropic with a narrow distribution of intensity in the transverse $[1\bar{1}0]$ direction (along the rods) around the (880) reflection and a very broad distribution along the [001] direction (across the rods). Moreover line profiles through the latter map showed a 3-peak structure. This is interpreted in terms of a small tilt or roll of rods with larger width. As with [001] oriented substrates[4], the tilt helps accomodate mismatch between the substrate and the epilayer for the broader rods. Narrower rods are assumed to be untilted. We are currently pursuing further analysis of the off specular reflections to explore distribution of strain in the two orthogonal in-plane directions.

The most significant results for the (111) oriented samples emerged from maps involving off-specular Bragg peaks. Figure 4 shows maps for samples of thickness 35 nm, 105 nm and 420 nm measured around the epilayer (10 2 6) reflection. The maximum in the intensity for the map of the 420 nm sample is centred around the position of the (10 2 6) reflection for bulk In_2O_3, indicating an almost completely relaxed film. By contrast the intensity in the map for the 35 nm sample is strongly displaced away from this position toward higher Q[111} and lower $Q[1\bar{1}0]$. This is indicative of a highly strained film with an increase in lattice spacings paralle to the surface and a contraction normal to it. From these results we have been able to derive a reliable new value for the Poisson ratio ν of In_2O_3 strained normal to the [111] direction. The value of ν=0.31 obtained in this way[5,6] is of critical importance in using strain to tune the bandgap of In_2O_3[7].

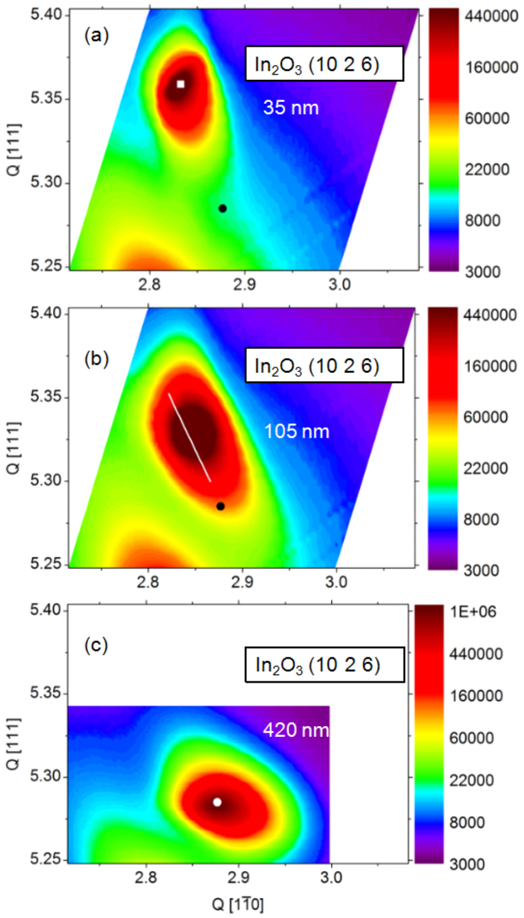


Figure 4. Reciprocal space maps around the In_2O_3 (10 2 6) epilayer reflection for the three samples of different thickness studied in the present work. The horizontal axis represents transverse wavevector transfer along the $[1\bar{1}0]$ direction while the vertical axis represents longitudinal wavevector transfer along the [111] direction, both in reciprocal lattice units of the substrate. The black spots in (a) and (b) and the white spot in (c) show the position in the map corresponding to bulk In_2O_3, while the white square in (a) identifies the position of maximum scattering intensity.

The film with thickness of 420 nm is almost completely relaxed while the film with thickness of 35 nm is highly strained. The intermediate 105 nm film appears to be strained close to the interface but partly relaxed away from the interface. The white line shown for this sample in panel (b) links the positions of maximum scattering intensity in transverse slices through the map and is seen to extrapolate toward the position for bulk In_2O_3.

In summary reciprocal space mapping experiments have provided valuable insight into the way lattice mismatch in thin films of In_2O_3 on Y-ZrO_2 is accommodated in differently oriented films.

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